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# 5<sup>th</sup> Historic Mortars Conference

## The initial reactions of lime-pozzolan pastes for conservation of masonry

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### Abstract

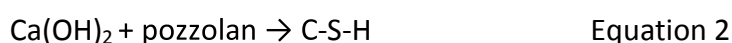
Throughout the lifetime of historic buildings the masonry will often undergo at least one conservation intervention. The need for conservation arises from the different elements of the building and to this end mortars have a very important role to play. Historic lime mortars and the various components from which they are made including the type of lime, pozzolan and aggregates have been extensively studied. They have received attention concerning their degree of reactivity and how they interact with the masonry units which are commonly stone or brick. The pozzolan-lime reactions, and the hydration and carbonation processes are important factors that influence the final properties of any lime-pozzolan mortars. In this study lime-pozzolan paste combinations are defined as a combination of calcium lime (CL 90) with a pozzolanic additive. The pozzolanic materials; wood ash, brick dust and Argical M1000 have been investigated in this study. To understand the role of the different pozzolans on the initial hydration reactions of the lime in pozzolan-lime pastes, the rheology and hydration kinetics were evaluated using a TA Hybrid Rheometer DHR – 2 and a Calmetrix I-Cal 4000 calorimeter respectively. In terms of kinetics evolution, Argical M1000, a type of metakaolin exhibited greater pozzolanic activity than wood ash and brick dust. Calorimetry and rheology tests have shown corroborative results regarding the initial hydration reactions of the pozzolan-lime pastes, different effects were observed for each type of pozzolan.

### 1 Introduction

With age materials are expected to fail, this failure of the macrostructure is very likely to have been caused by changes at the nano and microstructural level [1]. It has been demonstrated that there is a correlation between the chemical and physical properties of pozzolans and the performance at the micro scale within historic buildings [2]. The longevity of a repair made using these materials can be influenced by these interactions. It therefore follows that to be able to understand the behaviour and properties of historic mortars, it is important to understand the behaviour of the types of binder, aggregates and additives that have been used. In this study, a number of representative pozzolans were chosen to investigate their effects on mortar performance. Within the test mixes the proportions of CL90 hydrated lime, fine sand and water were kept constant.

Pozzolanic materials are siliceous or siliceous and aluminous in nature as defined in the ASTM C618 Standard [3]. They have no binding ability by themselves to form calcium silicate

hydrate (C-S-H) and calcium aluminate hydrate (C-A-H). To produce C-S-H or C-A-H pozzolanic materials require mixing with calcium hydroxide and water (Equation 1) [4]. Limes are classified as hydraulic and non-hydraulic (also called hydrated lime). Hydraulic limes can gain strength in the short term since they contain hydraulic phases which form calcium silicate hydrate (C-S-H) phases upon contact with water. These (hydraulic) phases originate from the reaction of lime with impurities containing silica, alumina and iron during the burning process at specific temperatures [5]. Non-hydraulic limes can be used in the form of lime putty, produced from quick or pure lime, or as a dry hydrated powder. However since non-hydraulic lime does not have any siliceous or alumina siliceous constituents, they require additives such as pozzolans to allow the formation of C-S-H phases during setting. Long term strength in limes is gained through carbonation as described by Equation 1 [6]. In this process carbon dioxide will dissolve in water forming carbonic acid which subsequently reacts with the lime forming calcium carbonate which leads to hardening (Equation 1). The pozzolanic reaction (Equation 2), hydration and carbonation processes (Equation 1) play a key role in determining the compatibility and durability factors for pozzolan-lime mortars used in conservation.



Previous studies have been carried out on the characterisation and standardization of mortars. In 1993 ICCROM (International Centre for the Study of the Preservation and Restoration of Cultural Property) and English Heritage carried out a joint study of the Smeaton Project and between 1997 and 2004, a study on pozzolanicity with cement was carried out by the British Standards Institute [7]. Any standardization for pozzolan-lime for use in renovation interventions has not been carried out since this time. This study provides new information on the behaviour of pozzolans. There are many studies [8-11] which investigate the physical characteristics of pozzolans such as surface area, particle size, fineness on the reactivity of lime-pozzolan pastes.

In this study we have investigated the physical and chemical characteristics of the pozzolans. The first step was the characterisation of raw pozzolanic materials. For the elemental analysis Energy Dispersive X-ray Spectroscopy (EDX) was carried out. X-ray Powder Diffraction (XRD) was used to identify the crystalline phases. The rheometer and calorimetry tests were performed to obtain information about the mechanisms of hydrated lime early hydration in the presence of pozzolanic materials. This study will investigate the effect of different pozzolan-lime combinations. The rheology of the pastes coupled with the oscillation tests provided information about how the behaviour of the different pastes change under the stress over time. This paper will look to correlate the kinetics of reactions with the changes in rheological behaviour. Together the hydration / pozzolanic kinetics and the monitoring of the setting behaviour of the pastes via the rheology studies will provide

information about the effect of the different pozzolans on the properties of the pozzolan-limes pastes.

## 2 Experimental Methodology

### 2.1 Materials

The lime selected for this study was Limbux CL90 hydrated lime supplied by Tarmac. It has low levels of impurities and does not contain any aluminates and silicate phases and therefore, does not have inherent pozzolanic activity. In this study three different pozzolanic materials have been investigated; wood ash, brick dust, and metakaolin (Argical M1000) supplied by Cornish Lime, Bodmin, UK. The pozzolan-lime paste mix proportions are presented in Table 1.

Table 1: Pozzolan-lime pastes mix proportions.

	% Pozzolan in Paste	Total mass (g)	Lime (g)	Pozzolan (g)	Water (g)	P/L* Ratio	B/W** Ratio
Mix 1	10%	50	5	20	25	0.25	1
Mix 2	18%	50	16	9	25	0.56	1
Mix 3	25%	50	12.5	12.5	25	1	1
Mix 4	34%	50	17	8	25	2.15	1

\*P/L: Ratio of Pozzolan/Lime \*\*B/W: Ratio of Binder (Lime + Pozzolan)/Water

Within the literature the pozzolan to lime (P:L) ratio of typical mixes range from 2.125 to 0.25. The most effective P:L was found to be 0.56 [12]. The ratios studied before such as 0.25, 0.56, 1, 2.15 for P/L ratio are chosen for this study as well.

### 2.2 Instrumentation

A Calmetrix I-Cal 4000 Calorimeter was used to obtain information about the kinetics of the reaction between the pozzolans and lime. The hydration reactions were analysed for a period of 120 hours to investigate the effects of the different pozzolans with the CL90 lime.

Microscopic analysis was carried out using a Joel JSM 6301F field emission scanning electron microscope with Energy Dispersive X-ray Spectroscopy (EDX) to obtain the elemental composition of the different raw materials. All samples were left under vacuum for 24 hours prior to testing to reduce moisture levels.

TA Hybrid Rheometer DHR-2 was used for the rheology studies. Changes in the paste rheology were analysed with viscosity when the stress is applied by time. A geometry gap of 1mm was utilised for all experiments. The upper and lower plates were disposable and had diameters of 25 and 60 mm respectively. The mixes were hand-stirred for 3mins before

placing on the lower plate. To reduce water loss via evaporation, the plate area was covered by a plastic lid, after the geometry gap is applied.

Characterisation of the crystalline phases present in the specimens was carried out using a WinXPOW PKS\_2.01 Version X-ray diffractometer. CuK $\alpha$ , X-rays of wavelength 1.540598Å were used in combination with a Multi-MYTHEN detector. Scans were carried out over a 2-theta range from 17° to 75° with a step size of 0.015° and a time per step of 300.0 Ms.

### 3 Results and Discussion

#### 3.1 Characterisation of raw materials

Argical M1000, Wood ash and brick dust are produced at temperatures of 600-800 °C, above 850 °C and above 1000 °C respectively. The elemental analysis (EDX) confirmed that CL90 is a pure hydrated lime which does not contain silicon (Si), aluminium (Al) or magnesium (Mg). The highest quantity of Si was observed in Argical M1000 which was 23.12% by weight. Cornish Lime brick dust (21.01%) had a comparatively lower Si weight percentage than Argical M1000. Wood ash showed the lowest Si and Al content which are important for promoting pozzolanic reactions with lime (CL90). The amount of aluminium oxide impacts the strength, setting characteristics, microstructure, acid resistance of the properties and metakaolin includes 40% aluminum oxide while other pozzolan, slag has 10% aluminum oxide content [13]. Walker and Pavia reported that the initial setting time of pozzolan-lime pastes are increased by the presence of silica and alumina phases, while the content of calcium and magnesium oxides decrease it [14]. Contrarily, Mg(OH)<sub>2</sub> is not associated with the strength of samples in terms of the studies of Edwards [15]. In terms of this study, Argical M1000 has the highest initial setting time while wood ash has the lowest regarding their silica and alumina contents. This is shown in Figure 6 where the lowest initial reaction delayed Argical M1000 and brick dust initiated. The presence of Ca as not detected for Argical M1000 while Cornish Lime brick dust and wood ash had levels of 18.15% and 21.65% respectively. Quantities of chlorine (Cl) were detected in the wood ash along with a high proportion of potassium (K). Small amounts of K and sodium (Na) were detected in the wood ash and brick dust, Figure 1.

The X-ray Diffraction Patterns (XRD) of the pozzolans, shown in Figure 2, were used to identify the crystalline phases present in the specimens. Quartz which was identified in the Argical M1000, brick dust and wood ash by peaks at 2-theta values of 20.85°, 26.63°, 36.49°, 50.17° and 68.18° [16]. In the wood ash, wollastonite was identified by peaks at 2-theta values of 28.07°, 34.58°, 36.49°, 42.46° , 59.95° [17], and potassium chloride through peaks at 28.414°, 40.618°, 58.792°, 66.568° [18], sodium chloride peaks at 31.569°, 45.155°, 56.098° [18].



Figure 1: Elemental composition of the raw materials (Weight %)

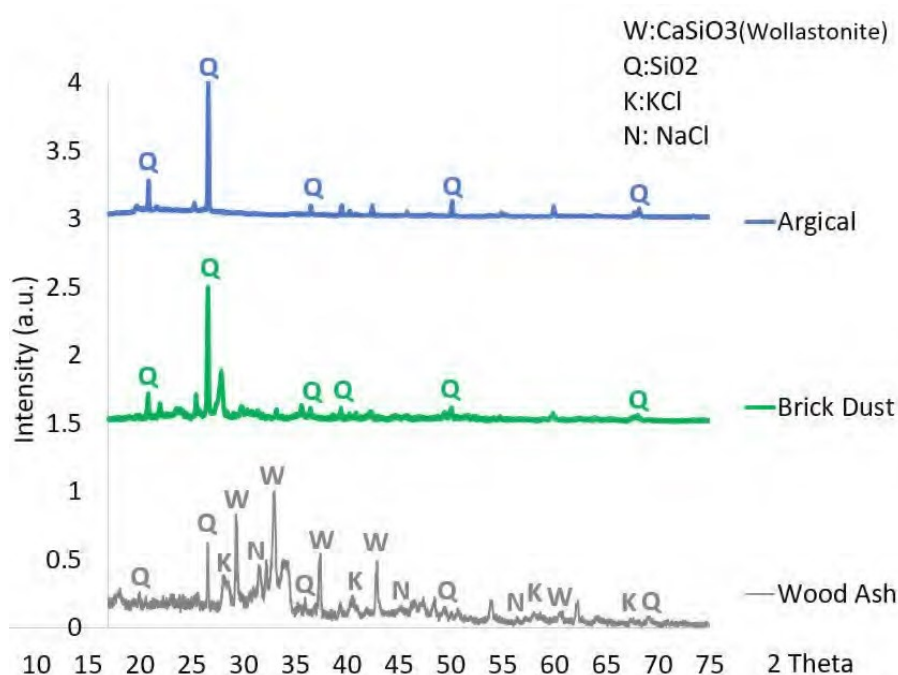


Figure 2: Representative Diffraction Patterns of raw materials

With the  $\text{SiO}_2 + \text{Al}_2\text{O}_3$  content, the pozzolans are rated since they are associated with the hydraulic components in limes [14]. Therefore, the type of pozzolan used in a mortar will have its own characteristic pozzolanic activity and acceleration period associated with hydration. The dissolution and hydration reaction are likely to occur earlier than the pozzolanic reactions. However the separation of these effects on the kinetics are not straightforward [19]. This study investigates the early kinetics within pozzolan-lime pastes.

### 3.2 Isothermal Heat Evolution Calorimetry

Since the correlation between the first day reactions and 28-day strength was found, the isothermal calorimetry test is studied to analyse the initial reactions [20]. The reaction kinetics were evaluated using isothermal calorimetry. The pozzolan-lime pastes (lime, pozzolan and water) were mixed for 3 minutes in plastic cups prior to loading into the chamber of the calorimeter. Measurements were recorded for 120 hours using a temperature of 40 °C. Previous studies by Chaudhari et al which compared results at temperatures of 25°C and 40°C indicated that better results were achieved at 40°C [20, 21].

The power (mW/g) and time(h) curves of mixtures showed endothermic and exothermic peaks (Figure 3, 5 and 6). The curves of lime with brick dust and lime with metakaolin-Argical M1000 showed small exothermic peaks (approximately 15.7mW), while the endothermic reaction is seen by the curves of lime with wood ash (-7.2mW) (Figure 6). The heat evolution for the initial hydration reactions (1.5 h) are shown in Figures 3 and 4. Any new phases are seen in the graphics. The difference between lime-gypsum-metakaolin and lime and metakaolin was studied by Tydlit et al [22]. This showed that lime with pozzolanic pastes exhibited a very small peak in the power versus time plots while gypsum additions accelerated the heat evolution two or three times more and produced another phases.

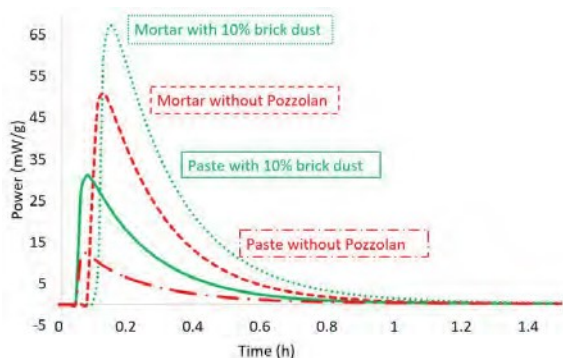


Figure 3: Power graph for lime mortars and pastes with and without brick dust (pozzolan). Measurements taken at 40 °C

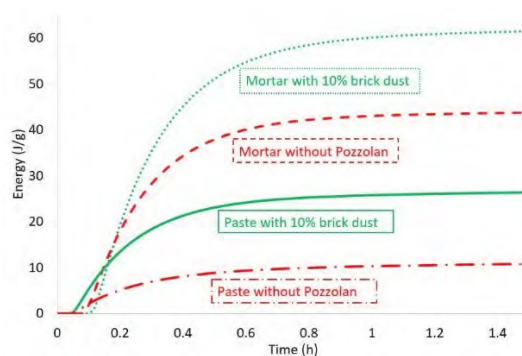


Figure 4: Energy graph for lime mortars and pastes with and without brick dust. Measurements taken at 40 °C

The curves in Figure 3 show the differences power output between mortar and paste with and without brick dust for experiments performed at 40 °C, while the curves in Figure 4 show energy differences between mortar and paste. As shown in Figure 3 and Figure 4 the power output and heat evolution are not as high for the pastes as they are for the mortars with the same pozzolan (brick dust). It is expected that pozzolanic materials would enhance the paste and mortar, however it was also observed that the addition of the sand increased the rate of heat evolution. Therefore, suggesting that sand could be improving the particles' dispersion in the mix, exposing the surface area of all the constituents and therefore allowing more reactions to take place (Figure 4). The peaks in Figure 3 and Figure 4 might be accelerated by the physical effects with unreactive components such as sand.



In Figure 5 it can be seen that the mix with the higher percentage (34%) of Argical M1000 delivered the highest power output. For the lime paste without Argical M1000, it is clearly seen that after the first energy peak around 0.2 hours, the power output decreases for all the samples. However, Argical M1000 contents of 25% and 34% presented slight increases. This suggests that more reactions take place with the increase in the Argical M1000 content and at 40°C, the rates of heat evolution are directly proportional with the quantity of pozzolans (Figure 5). Any new phase within 5 days was seen after the first peak but there was a wide shoulder shape which may be explained by formation of a new phase for 34% Argical M1000 content of paste.

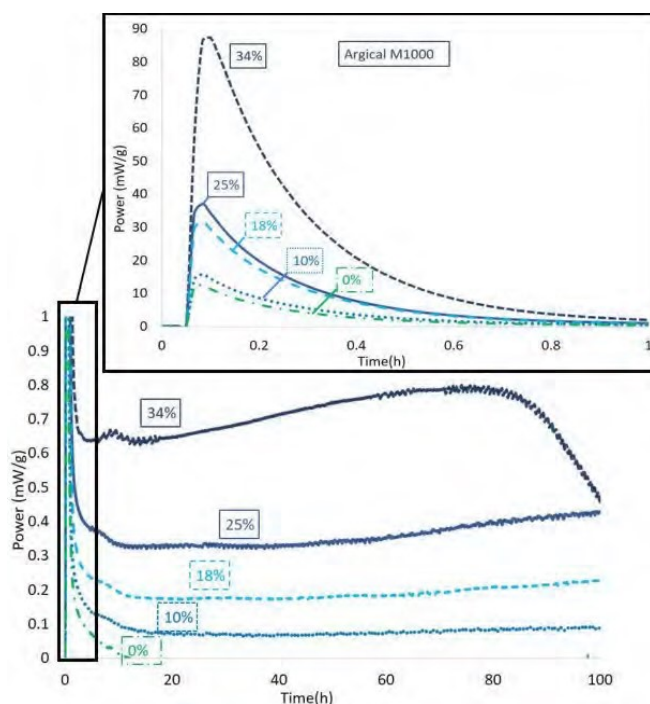


Figure 5: The reaction kinetics of CL90 lime pastes containing 0, 10, 18, 25 and 34% Argical M1000 at 40 °C

Figure 6 shows a comparison of the power output of different types of the pozzolan at the same percentage within the typical water and lime quantity as binder water ratio (B/W) is 1. Different kinetics concerning the dissolutions and reactions are shown. Argical M1000 showed a highest energy output, suggesting a high pozzolanic activity while wood ash showed the lowest energy output. Therefore, the heat evolution of the different pozzolan-lime pastes highlighted the significant effect that the type of pozzolan can have on the kinetics.

EDX analysis of the ash indicated 0.41% chlorine and the alkali metals sodium and potassium. Peaks for potassium chloride and sodium chloride were also observed in the XRD pattern (Figure 2). As the dissolution of these salts is endothermic their presence could be an explanation for the negative power values recorded for the wood ash compared to the Argical M1000 and brick dust which showed exothermic reactions.

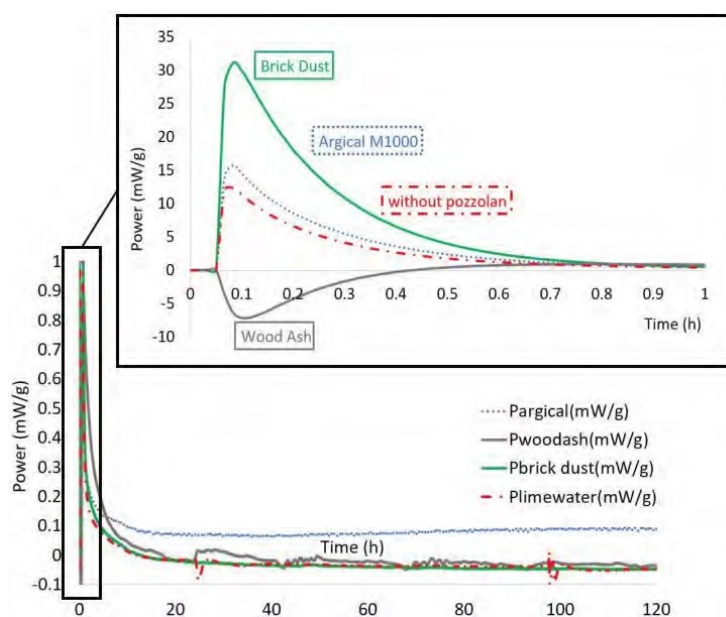


Figure 6: The kinetics evolutions of the pastes with different Pozzolans 10% at 40 °C

For example, even though the evolution of brick dust is the highest for the first 40 hours, Argical M1000 accelerated the energy evolved to a greater extent compared to the other pozzolans (wood ash and brick dust) after the 40 hours mark (Figure 7).

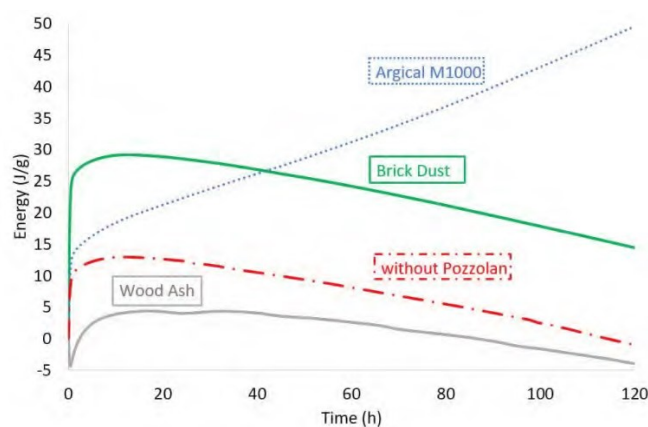


Figure 7: The energy kinetics evolutions of the pastes with different pozzolans 10% at 40 °C

The heat evolutions between 0.2 h to 1 h of all mixtures shows similar decreasing behaviour. Some pozzolanic materials appear to show activity later on. Therefore, more research is required to monitor the pozzolan-lime pastes for a longer period of time.

### 3.2.2 Rheology of the pozzolan-lime paste

The Rheology gives information about the compositions in terms of their flowability, behaviours under pressure, shear rate, resting times which are necessitated indirectly for conservation. The pozzolan–lime mixes were studied using a rheometer which applies a force to materials over time to provide information on the flow and deformation behaviour.

The shear stress and shear rate proportions are calculated to measure viscosity of the materials to define the characterisation with flow curve. The results presented in Figures 8 to 11 show that all pastes have exhibited a pseudoplastic behaviour (non-Newtonian) because of the shear thinning characteristics exhibited. For the mortar application, high viscosity and plasticity are sought to be achieved [23] since the viscosity of pozzolans shows the workability. The pozzolans used in this study are listed as their workability respectively; Argical M1000, wood ash and brick dust (Figure 13). Viscosity of all samples is increased over time in Figure 10 as it is observed to be changing from liquid like behaviour to solid like behaviour. The increase of yield stress of Argical M1000 separated from others is related to the workability of the paste [24, 25] (Figure 11). However, it is not correlated with the kinetics of energy evolution of the hydration reactions, which showed a higher early energy out for brick dust.

The graphics between Figures 11 to 13 show oscillation tests of three pozzolans at 40 °C. Due to Argical M1000 consisted paste being more structured than the other pozzolans a more elastic behaviour with the higher internal structure is shown. The storage and loss modulus of Argical M1000 pastes show behaviour changes from an initial elastic and liquid like behaviour then stiffening and changing to a solid like behaviour over time. Figure 10 shows that the materials with 90° phase angles at the beginning are showing more liquid like behaviour. The more solid like behaviours characteristic of 0° angles are observed later. This behaviour is expected when the loss of water and hydration reaction in 1.5 hours are considered. However, Argical M1000 paste is becoming a more structured form with less oscillation force compared to the past containing brick dust.

However, the tests show promise that the rheology measurements can be used to study the initial changes in stiffness when the pozzolans are first mixed with lime. This could be due to pozzolanic reaction, dissolution and water absorption into porous particles, such as the brick dust.

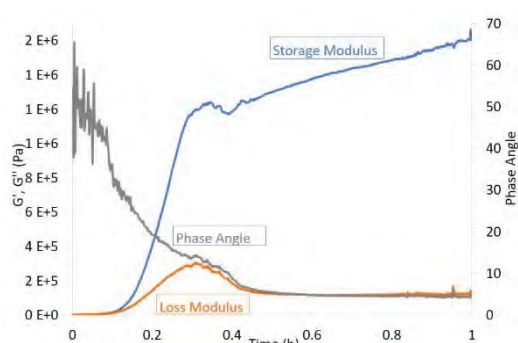


Figure 8: Storage modulus, loss modulus and phase angle against time for 10% Argical M1000 at 40 °C

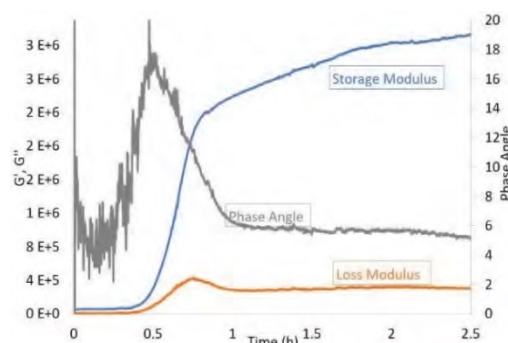


Figure 9: Storage modulus, loss modulus and phase angle against time for 10% Brick Dust at 40 °C

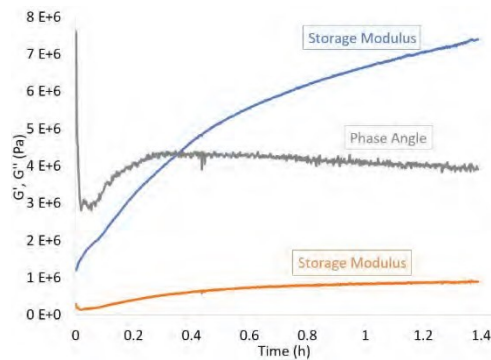


Figure 10: Storage modulus, loss modulus and phase angle against time for 10% Wood Ash at 40°C

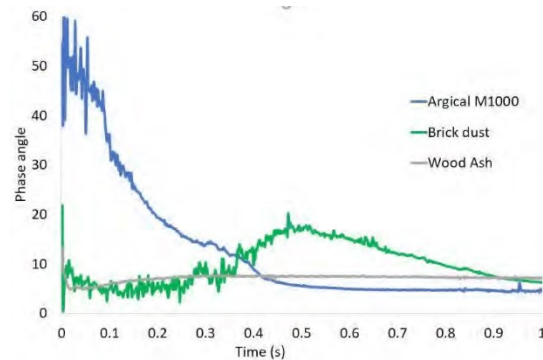


Figure 11: Phase angle against time for Argical M1000, brick dust and wood ash

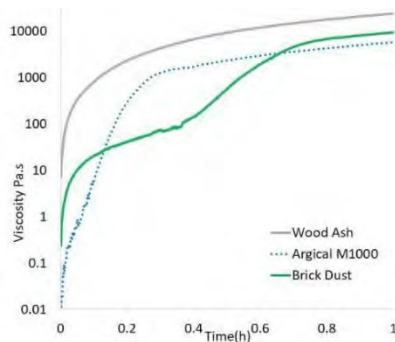


Figure 12: Viscosity against time for Argical M1000, brick dust and wood ash

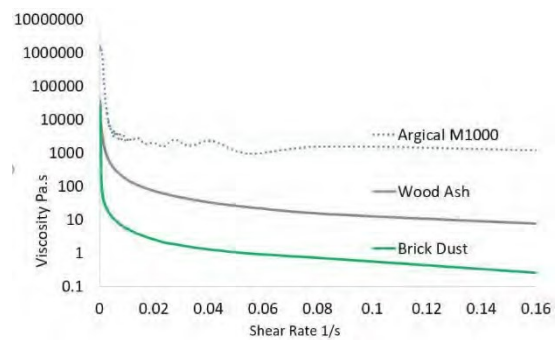


Figure 13: Viscosity against shear rate for Argical M1000, brick dust and wood ash

## 4 Conclusions

The early reactions between lime and Argical M1000, wood ash and brick dust were studied using calorimetry and rheology. Since Natural Hydraulic Limes from different manufacturers can exhibit varying properties, even within the same classification as defined by standard EN459, there is a growing interest in alternative mortar systems.

Calcium lime – pozzolan mortars offer an alternative and here we evaluate the performance of such mortars through the study of rheological and calorimetry properties [26]. When pozzolanic limes are used in conservation the rheological properties are important as they are related to properties such as workability which is important for practitioners. The study helps in conservation applications in terms of understanding the materials and their reactions.

The following conclusions have been drawn:

- The heat of reactions evolved when Argical M1000 was mixed with lime increased with the amount of Argical M1000 within the first 1 hour after mixing. The evolution of all heat peaks was completed within 1.5h.
- After the first peak between 0.1 to 0.6h, the mixture consisting of 34% and 25% Argical M1000 showed little changes which can be new phases in progress.

- The peaks of the kinetics evolution ascended directly proportionally with the amount of Argical M1000 (34%, 25%, %18, 10%) in the mixtures. Hence, it can be claimed that the pozzolan effected the heat evolution of mixtures in terms of both acceleration and increase.
- After 1 hour the heat generated from the reaction between lime, pozzolan and water was not detectible. No further heat was detected by the calorimeter during a further 120h of monitoring.
- The reaction rate of CL90 lime paste with the pozzolans; brick dust and Argical M1000 is accelerated compared to the reference sample lime pastes without pozzolan.
- The ability of sand to accelerate the pozzolanic reaction can be observed by comparing the mortars with and without pozzolan where the heat evolution was increased by the presence of sand. It is also noteworthy that the hydration reaction was delayed.
- Energy dissipation and viscosity of material behaviour are seen corresponded with the kinetics evolution of the pozzolanic paste in isothermal calorimetry. When the hydration reaction is decelerated (0.2-0.6h), the viscosity and stiffnesses are changed for Argical M1000. For brick dust the changes of rheology are started after the hydration reaction. However, for wood ash, the time of the kinetic changes on calorimetry was approximately at the same arrangement (0.1-0.6h) with the rheology.

## 5 Future work

Future work will involve correlating the minerology of the pozzolans with the calometry and rheology data. We intend to investigate how the dissolution of minerals in the pozzolans affects the reactions.

The physical parameters such as surface area, fineness and particle size distribution will then be determined and fixed for all materials to enable a more accurate comparison of the chemical parameters.

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